A UNIVERSAL SET-UP FOR THE MEASUREMENT

OF THE THERMAL PROPERTIES OF LIQUIDS AND SOLIDS

By

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ABSTRACT

A modified apparatus for the measurement of the thermal activity (b), diffusivity (a), conductivity (λ)and volumetric heat capacity (ρC_p)coefficients of noncoducting and conducting liquids and solids(thick and thin layers) is described.

In case of liquids (melts) a thin platinum wire of 15-50 μm diameter ,fixed by two stainless steel electrodes,was immersed in the investigated medium in a stainless steel cell. The wire was connected as a fourth arm of an AC bridge.The third harmonic signal generated across the diagonal, was amplified,filtered and measured at two(or more) frequencies along with power dissipated in the wire.Then the obtained experimental data have been processed by means of a dynamic interactive program with personal computer and the mentioned thermal properties were calculated.The described technique has several advantages ,the most important of which is that convection is highly suppressed, the role of radiation has been treated theoretically and adequately considered as described inan earlier work.

The described set-up was tested with water and toluene as standard liquids. The thermal properties of $AgNO_3$ and $Cd(NO_3)_2$ are reported .

The describeed technique (strip and wire)was applied for measurement of the mentioned thermal proprties of thick and very thin layers of nonconducting solids ,with assured perfect thermal contact. Theory of this technique and results for thin plane and cylindrical layers are to be published in future works: parts II and III ofthis work.

When conducting liquids (salts or melts) were investigated ,a wire coated with a very thin nonconducting layer was used.Theory of these techniques and results are to be published in future works : parts IV and V of this work.

Key words: AC-heated, wire, set-up, thermal properties, water,toluene,NaNO3, $Cd(NO_3)_2$.

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1.Introduction

Several techniques have been used for the measurement of thermal properties of liquids and melts [1-8]. Most of these techniques suffer from the interference of convective and radiative heat transport ,along with the conductive transport.The most important advantage of the AC-heated wire technique is that the temperature oscillations of the metallic wire are confined to a very thin layer of the investigated liquid. Consequently, the convective heat transfer is supressed.The role of the radiative heat transfer is considered[9].

The AC-heated wire technique was successfully used for the measurement of the thermal activity, diffusivity ,conductivity and volumetric heat capacity coefficients of different liquids and some solids at high temperatures and pressures [1], [10].

In this work, a modified set-up has been built.A detailed analysis of all the components of the electric circuit was carried out.With the help of a a specially written PC program,the mentioned thermal properties were calculated.The set-up,after being tested with water and toluene as standard liquids, was used for measurement of the mentioned properties of AgNO3 and $Cd(NO_3)_2$.

2.Experimental

2.1.Description of the experimental set-up

A ceramic(or a stainless) experimental cell(1x1.8x13cm) Fig.(1) was filled with the investigated liquid and then covered by a ceramic lid, with a platinum wire(of 15,50 μm diameter ,and length 8-10 cm), fixed in place by means of two stainless steel electrodes.

The cell was then tightly sealed with silicon rubber and mounted inside an electric furnace that provides the mean temperature of the sample, and powered by a 10 kW variac connected to the mains through an insulation transformer . The experimental cell and the electric furnace were then mounted in a water-cooled metallic chamber.

The metallic chamber was placed on a metallic base of diameter 40 cm, that contains all the electric leads, Fig.(2). The chamber and base were evacuated and then filled with nitrogen gas at atmospheric pressure from a nitrogen cylinder Fig.(3).

Finally the platinum wire was connected as one arm of an AC-bridge that consists of two precise shielded decade resistance boxes (0-111111 Ω). The fourth arm was a 5 Ω standard resistance. The bridge was fed by an AC-signal generated by a signal generator and an audio amplifier. The signal frequency was measured by a frequency meter.When the bridge was completely balanced at a frequency f(60-120 Hz),which was judged by the shape of the signal from the diagonal of the bridge on the screen of an osiclloscope , the third harmonic signal (3f) was amplified ,filtered by a tuned filter and displayed on the screen of another identical oscilloscope. For more control the output signal of the amplifier was displayed on the screen of the same oscilloscope. Both the third harmonic signal amplifier and filter were built with the same IC .Then the third

harmonic signal was adjusted to be enveloped by two horizontal lines generated on the second channel of the second oscilloscope from another function generator.Then the amplitude of this signal($E_{3\omega}$) was measured by means of an accurate multimeter .

Readings of multimeters of the voltages across the bridge (V_T), across the standard resistance (V_S), across the platinum wire (V_r) and $E_{3\omega}$ were recorded, along with the values of the readings of the decade resistance boxes.

The amplitude of the third harmonic $E_{3\omega}$ was then accurately measured by means of a potential divider with a ratio 2 /1000 that consisted of two precise shielded decade resistance boxes(0-111111 Ω).

It is worth to mention that grounding of one side of the AC-bridge leads to the generation of the second harmonic temperature oscillations.This point has to be investigated.The advantage of this modification is that the temperature oscillations signal is about twice that of the third harmonic one.

2.2 Measurement of power

As has been described , measurement of power was carried out with multimetrs connected across the standard resistance and the wire.More-over ,a detailed analysis of the circuit was carried out to consider the internal resistance of the power amplifier as well as the output amplifier ,in addition to all other elements of the circuit ,like the capacitance box . This proved to be quite essential to get correct values of the temperature oscillations amplitude and power.

2.3 Calibration of the third harmonic signal

Calibration of the third harmonic signal has been done in two ways; either by means of a potential divider or by means of the AC-bridge itself.

2.3.1 The potential divider method

In this method ,a signal of triple frequency is fed to the input of a potential divider with ratio 2/1000, that consisted of two precise shielded decade resistance boxes(0 - 111111Ω).The output signal was then applied to the input of the amplifier(1),filter , oscilloscope (1) ,to digital multimeter (3).The amplitude of the input signal is adjusted to get the same output signal ,obtained during measurement.This has been repeated for each experimental point keeping the measuring circuit connected.

2.3.2 The AC-bridge method

In this method ,the cell is disconnected ,and substituted by a decade resistance box ,an equivalnt resistance is set , the AC bridge is fed with a signal of triple frequency , and the bridge is balanced,with the capacitor box disconnected.Then the bridge is unbalanced to get a signal equal to the measured one.This signal is then accurately measuredby means of digital multimeter(3). Solving the bridge circuit ,the third harmonic signal on the wire was calculated as part of the program that processes the obtained experimental data .

2.4.Measurement of the temperature coefficient of the platinum wire

The coefficient of change of resistance of the platinum wire with temperature has been measured in a separate experiment . The experimental cell was put in an oven with regulated temperature control ,keeping the temperature constant within + 0.1 C. The temperature of the wire was measured by means of a digital thermometer with 0.1 C error. This has been performed for each wire.

3.Processing of the experimental data

In[2] it was shown that the reduced temperature oscillations amplitude of the wire is related to the thermal properties of both the wire and the investigated medium through the following relation:

$$\theta/\theta_o = \left[\frac{her^2(\chi) + hei^2(\chi)}{(\eta\, hei(\chi) - her`(\chi))^2 + (\eta\, her(\chi) + hei`(\chi))^2} \right]^{1/2}$$

where

$\theta_o = Wo/\,Sb\sqrt{2\omega}$, $x = m'C'\sqrt{2\omega}/\,Sb$, $\chi = \sqrt{2\omega/a}\; r$, and

θ - amplitude of temperature oscillations of the wire,S-side area of the wire, r- radius of the wire,m: mass of the wire, C′: specific heat of the wire material, her,hei,her′, and hei′:Kelvin functions.

A specially written program was used to process the experimental data to determine the mentioned thermal properties. The measured f, V_T, V_S, V_r and $e_{3\omega}$ at different frequencies (60-120 Hz) and parameters of the used platinum wire were fed into PC. Processing of the data obtained for two frequencies (or more) for calculating the values of b, a, λ and ρC_p involving an iteration technique. Such a method saved time and effort, and improved the accuracy of the obtained results

More-over ,this approach simplifies the experimental technique considerably ,as there is no need to do measurements with a strip to complement the experiment.It has also been found that errors in measuring length of the wire, coefficient of change of resistance, are automatically reduced in the iteration technique because they identically affect both values of the temperature oscillations amplitude atboth frequencies.

4.Results and discussion

Calibration of the described set-up has been carried out with twice distilled water. The obtained values of b, a, λ and ρC_p for water were compared with data recommended in [8]. It was found that data for (b) is lower by 3% in the temperature range 30-90C. Data for (a) are lower by 2% at t=30C and higher by 3% at t=90C. For (ρC_p) our data is lower by 3% at t =30C, but at t=90C, the

discrepancy is less than 1%. Our data for (λ) coincides with the mentioned recommended data at t =30C, at t =90 C, they are lower by 1%.

Table I : Thermal properties of $AgNO_3$[10]

(C)	$ax10^7$ (m^2/s)	10^{-6} x ρC(Wkg/m^4.C)	λ (W/m.C)
50	2.1	1.48	0.31
60	1.8	1.59	0.29
70	1.79	1.62	0.29
80	1.69	1.71	0.29
90	1.60	1.81	0.29
100	1.55	1.87	0.29
110	1.50	1.93	0.29
120	1.47	1.97	0.29
130	1.44	2.04	0.29
140	1.47	1.97	0.29
150	1.48	1.95	0.28
160	1.68	1.72	0.28
170	1.27	1.12	0.14
180	1.63	1.22	0.21
190	1.77	1.24	0.15

DSC analysis of a specimen showed that this salt has an orthorhombic to to trigonal transition at 169.7C .The specimen melts at 216.9C.During cooling the mentioned transition does not take place.in table I are given the smoothed results for thermal diffusivity(a),volumetric heat capacity (ρC) and thermal conductivity coefficient (λ). The values of these properties show that the investigated temperature range is above the Debye temperature ,and the heat conduction is due to phonons.

Table II : Thermal properties of $Cd(NO_3)_2$[10]

t (C)	ax10^7 (m^2/s)	ρCx10^{-6}(Wkg/m^4.C)	λ (W/m.C)
35	1.43	2.24	0.31
40	1.62	2.28	0.36
45	1. 65	2.20	0.36
50	1.81	2.41	0.43
55	4.20	2.91	1.21
60	1.79	2.72	0.413
65	1.81	2.58	0.464
70	1.71	2.55	0.431
75	1.62	2.52	0.403
80	1.52	2.50	0.375

DSC analysis of a specimen showed that this salt starts melting at 55.6C and melts completely at 65.1C.A striking fact about this salt is that during cooling to the room temperature ,it does not solidify for several days.The same was observed for some other salts ,$Pb(NO_3)_2$.
In table II are given the smoothed results for thermal diffusivity(a),volumetric heat capacity (ρC) and thermal conductivity coefficient (λ). The values of these properties show that the investigated temperature range is above the Debye temperature ,and the heat conduction is due to phonons in this salt too.

The AC-heated wire technique has the advantage that the role of convection is suppressed because the investigated layer in the used frequency range was very thin (fractions of a millimeter). Moreover, the absence of convection was judged by the amazing stability of the third harmonic signal on the screen of the oscilloscope and the balance of the bridge. The role of radiation (photon transport)has been calculated according to the relations obtained in [8] and found to be negligible (O.O2-O.O5 %).
It is important also to mention that in case of a stainless steel measuring cell,the metallic wire used in the experiment was coated with a very thin nonconducting layer.This affected appreciably the experimental results.For this reason ,the internal surface of the stainless steel cell was coated with a thin nonconducing resistive layer.This phenomenon is currently investigated.

4.Conclusion

Due to the above mentioned advantages of the AC- heated wire technique , we can recommend it as an accurate and at the same time simple one for the measurement of the termal diffusivity, specific heat capacity, and thermal conductivity coefficients of liquids (and solids-bulk and thin layers as well) in a very wide range of temperatures and pressures.

5. References

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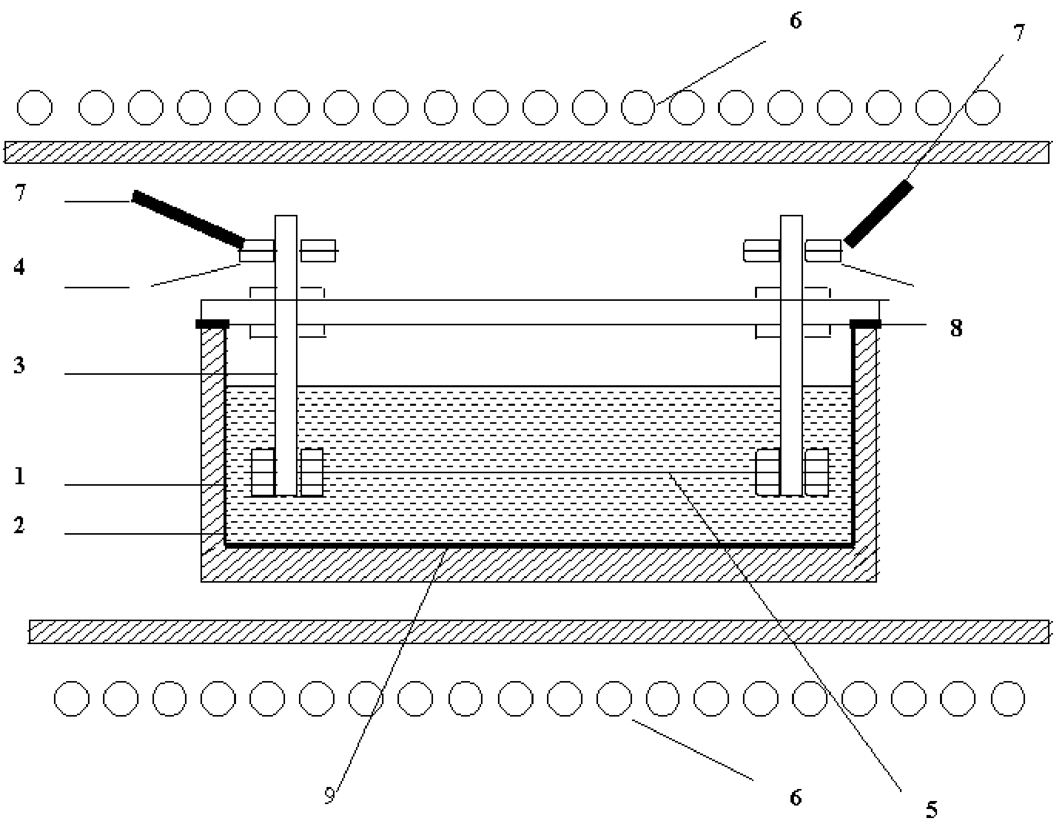
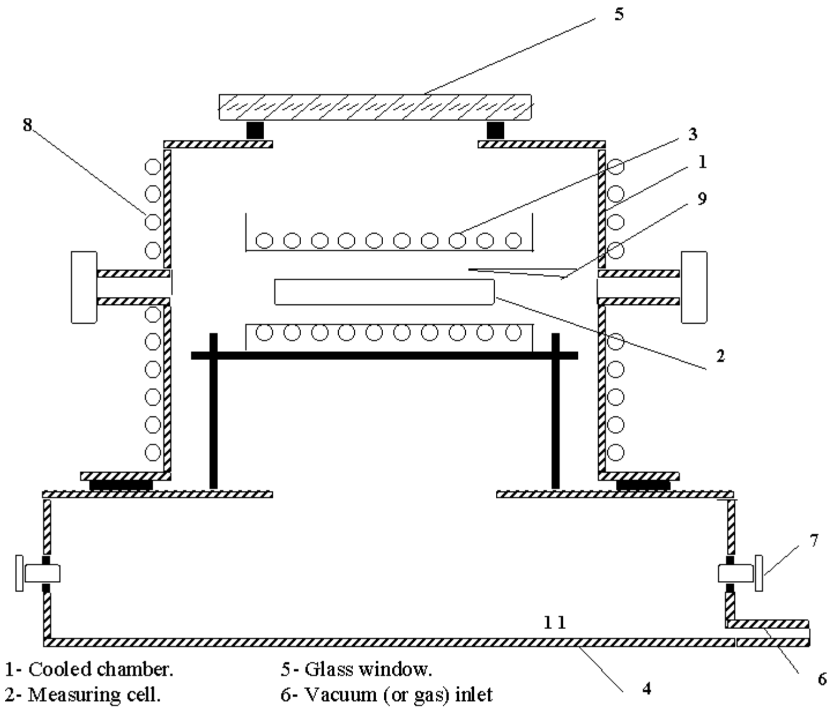


1- Ceramic (or stainless steel) cell.
2- Sample.
3- Stainless steel electrode.
4- Potential leads.
5- Platinum wire.
6- Furance.
7- Current leads.
8- Silicon rubber.
9- Resistive coating
Fig. (1) The Measuring Cell.



1- Cooled chamber.
2- Measuring cell.
3- Furnace.
4- Cylindrical bacse.
5- Glass window.
6- Vacuum (or gas) inlet
7- Electrical leads.
8- Cooling tubes.
9- Thermocouple.

Fig. (2) The Metallic Chamber.

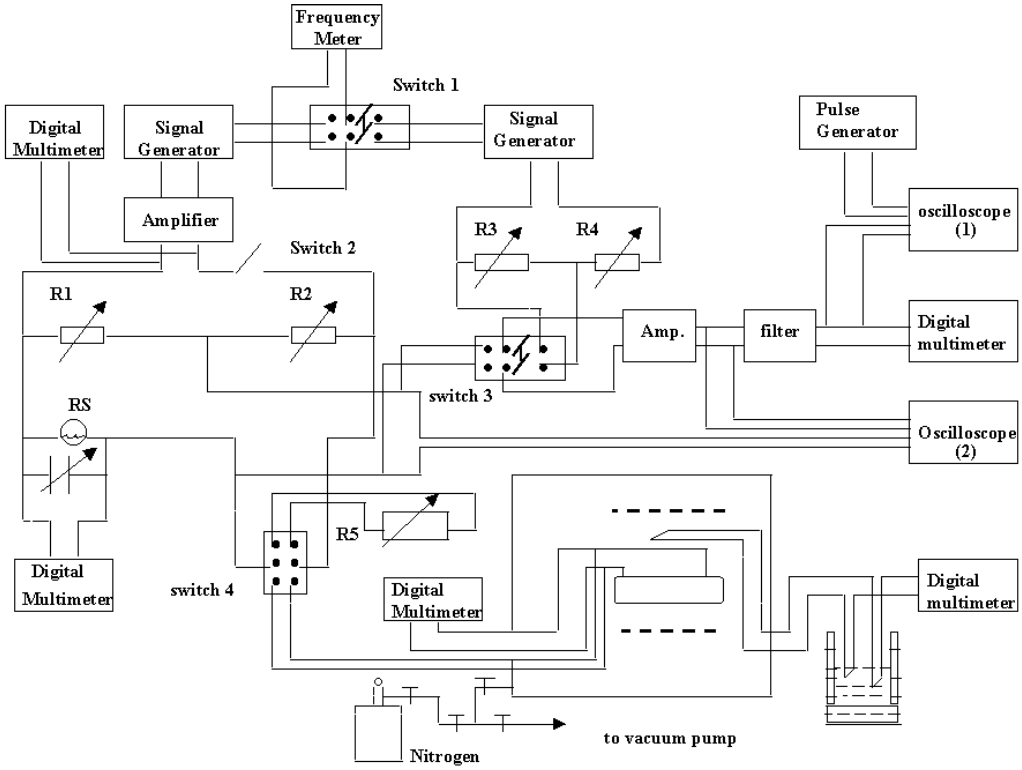


Fig. (3) Block Diagram of the Experimental Set-up